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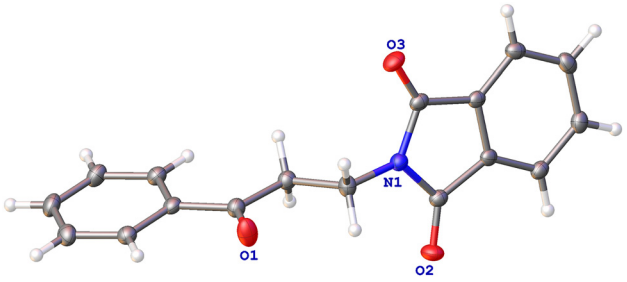
Synthesis and crystal structure of 2-(3-oxo-3-phenylpropyl)isoindoline-1,3-dione, $C_{17}H_{13}NO_3$



https://doi.org/10.1515/ncrs-2023-0342
Received July 22, 2023; accepted August 17, 2023;
published online September 5, 2023

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.16 × 0.14 × 0.12 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	0.78 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	73.8°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5078, 2663, 0.119
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2298
$N(param)_{refined}$:	190
Programs:	Olex2 [1], Bruker [2], SHELX [3]

Abstract

$C_{17}H_{13}NO_3$, monoclinic, $P2_1/c$ (no. 14), a = 9.5306(2) Å, b = 6.1971(2) Å, c = 23.3849(7) Å, β = 101.445(3)°, V = 1353.70(7) $Å^3$, Z = 4, $R_{gt}(F)$ = 0.0644, $wR_{ref}(F^2)$ = 0.1909, T = 120 K.

CCDC no.: 2289192

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

The 1-phenylprop-2-yn-1-ol (0.5 mmol) and isoindoline-1,3-dione (1.5 mmol) were dissolved in toluene (4 mL), and Ag_2CO_3 (0.075 mmol) and 2,3,4,6,7,8,9,10-octahydropyrimido [1,2-α]azepine (2 mmol) were added to the mixed solution for reflux reaction at 363 K for 12 h. After the reaction is completed, the mixture is extracted three times with ethyl acetate, the organic layers are merged, dried with anhydrous sodium sulfate to remove water, filtered to obtain the organic layer, and concentrated. Rapid column chromatography on silica gel (Petroleum ether: ethyl ester = 5: 1, v/v), and recrystallized with methanol to obtain pale yellow needle crystals.

2 Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Phthalimide and its derivatives are important structures in medicinal chemistry and organic chemistry [4, 5]. Phthalimide-based drugs have been successfully repurposed for erythema nodosum leprosum, multiple myeloma and myelodysplasia [6]. *N*-(acyloxy)phthalimides (NHPI esters) have emerged as suitable precursors of the corresponding carbon centered radicals under reductive decarboxylative conditions [7–9]. Therefore, the synthesis and modification of phthalimide has been a hot topic.

The basic structure of the title compound is a phthalimide derivative, containing a phthalimide and a phenylacetone. The bond lengths and angles from the title structure are within the normal range, consistent with those previously reported in the similar structure [10–12]. There are

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.66334 (17)	−0.1034 (2)	0.46288 (7)	0.0390 (4)
O2	0.35215 (15)	0.5431 (2)	0.41648 (6)	0.0312 (4)
O3	0.70904 (14)	0.4709 (2)	0.31323 (6)	0.0307 (4)
N1	0.54492 (15)	0.4637 (2)	0.37370 (6)	0.0221 (4)
C1	0.84965 (17)	0.0300 (3)	0.53629 (7)	0.0225 (4)
C2	0.86915 (18)	−0.1714 (3)	0.56318 (8)	0.0253 (4)
H2	0.809668	−0.288787	0.547528	0.030*
C3	0.9746 (2)	−0.2017 (3)	0.61259 (8)	0.0306 (4)
H3	0.987496	−0.339592	0.630602	0.037*
C4	1.0616 (2)	−0.0302 (3)	0.63578 (8)	0.0312 (5)
H4	1.133812	−0.050574	0.669712	0.037*
C5	1.04260 (19)	0.1707 (3)	0.60923 (9)	0.0305 (4)
H5	1.102275	0.287837	0.624913	0.037*
C6	0.93649 (18)	0.2011 (3)	0.55971 (8)	0.0262 (4)
H6	0.923361	0.339325	0.541864	0.031*
C7	0.73344 (19)	0.0535 (3)	0.48336 (7)	0.0241 (4)
C8	0.70255 (18)	0.2742 (3)	0.45596 (7)	0.0236 (4)
H8A	0.790176	0.332474	0.444926	0.028*
H8B	0.673377	0.374111	0.484511	0.028*
C9	0.58384 (19)	0.2580 (3)	0.40220 (8)	0.0256 (4)
H9A	0.614702	0.158636	0.373938	0.031*
H9B	0.498066	0.194683	0.413610	0.031*
C10	0.42799 (18)	0.5867 (3)	0.38238 (7)	0.0217 (4)
C11	0.41576 (16)	0.7686 (3)	0.34028 (7)	0.0205 (4)
C12	0.31797 (18)	0.9352 (3)	0.33008 (7)	0.0235 (4)
H12	0.243342	0.947361	0.351458	0.028*
C13	0.3328 (2)	1.0847 (3)	0.28732 (8)	0.0265 (4)
H13	0.266970	1.201028	0.279038	0.032*
C14	0.4436 (2)	1.0656 (3)	0.25636 (8)	0.0286 (4)
H14	0.452107	1.170100	0.227516	0.034*
C15	0.54208 (18)	0.8963 (3)	0.26693 (7)	0.0253 (4)
H15	0.617284	0.883088	0.245888	0.030*
C16	0.52517 (16)	0.7487 (3)	0.30940 (7)	0.0206 (4)
C17	0.60796 (17)	0.5499 (3)	0.33014 (7)	0.0222 (4)

three keto-groups in the structure with the distances 1.223(2) Å (C7–O1), 1.208(2) Å (C10–O2), 1.215(2) Å (C17–O3), respectively. The three bonds, in which the N atom participates, are 1.452(2) Å (N1–C9), 1.398(2) Å (N1–C10), and 1.388(2) Å (N1–C17).

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: Guizhou Administration of Traditional Chinese Medicine. (No.QZYY-2021-008). Guizhou Provincial Basic Research Program(Natural Science)(Qian Ke He Ji Chu-ZK[2023] Zhong Dian 047, Qian Ke He Ji Chu-ZK[2021] Yi Ban 561).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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